

## THE MORPHOLOGY AND THERMAL BEHAVIOUR OF TREATED AND UNTREATED SISAL FIBRE-EPOXY RESIN COMPOSITES

WILSON WEBO<sup>1</sup>, LEONARD MASU<sup>2</sup> & MAINA MARINGA<sup>3</sup>

<sup>1,2</sup>Faculty of Engineering and Technology, Department of Mechanical Engineering, Vaal University of Technology,  
Vanderbijlpark, South Africa

<sup>3</sup>Faculty of Engineering and Technology, Department of Mechanical and Mechatronics Engineering,  
Central University of Technology, Bloemfontein, South Africa

### ABSTRACT

*The influence of the chemical therapy of sisal fibres during the enduing procedures of mercerization (alkali treatment), silane handling and ultimately acid hydrolysis on the morphology and thermal behaviour of sisal fibre-epoxy resin composites were examined. Scrutinizing electron microscopy examination indicated the chemical handling procedures led to a reduction in the transverse dimension of the cellulose cells and an attendant reduction in the cross-sectional dimensions of the sisal fibres.*

**KEYWORDS:** Morphology, Thermal, Silane, Mercerization & Epoxy

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### 1. INTRODUCTION

Natural fibres were noted as biodegradable and naturally approachable sources of raw material for the fibre-reinforced composite industry (1, 2). Over the years, numerous studies were undertaken to separate cellulose from number of cellulosic fibre sources such as kenaf and ramie, bamboo, flax, sisal and rice straw (3-10). It has been reported that South Africa alone produces 2000 tonnes of sisal annually (11). The sisal producing areas in South Africa are: Limpopo province (Mokopane, Polokwane, Giyani and the Phalaborwa corridor); Kwa Zulu Natal province (Hluhluwe, Mtubatuba and Port Shepstone) and Northwest province (Madikwe) (12). Over the typical content of sisal fibres 67%-78% cellulose, 10.0% - 14.2% hemicellulose, 8.0%-11.0% lignin and 10% Pectin (13).

Imam et al. (14) prepared composites from Poly Vinyl Alcohol (PVA) and organic waste and further characterized those. Thermogravimetric analysis of the PVA-organic waste composites revealed mulch films in fields in them at higher temperatures. A research on the influence of natural fibres over the thermal and mechanical elements of PVA was carried out using DSC and DMA. It revealed the utilization of natural fibres as fillers might expand the range of implementation for PVA goodsto high temperature (16). It was also shown in the study that inclusion of extra 20 wt% of natural fibre raised the stocking modulus of neat PVA from 293MPa to 1034 MPa, an increase of 253%. Gregorova *et al.* (18) showed an enhancement of the thermal and mechanical elements of PVA strengthening along the treated Stika spruce fibres.

To expand their industrial implementations, natural fibres should tackle the limitation of incompatibility with hydrophobic polymer matrices in order to improve their dispersion within the polymer matrix, and also to increase the transfer of stress between them and the matrix. To decrease the hydrophilic feature of natural fibres, and to further enhance the adhesion effects, the fibre surface must be chemically modified. In this vein, several methods were analyzed using anhydrides, isocyanates and organosilanes(17).

This method of vacuum infusion is taken in this research so as to take advantage of its benefits of reducing porosity in the composites manufacturing. The method also aids correct wetting of the strengthening fibres and, enables way to finer and well built fibre/matrix interfacial connection and the attendant improvement in the mechanical effects of the fabricated composite (15).

From this paper, the influence of chemical handling on sisal fibres is investigated. The influence of this handling on the morphology of both treated as well as untreated sisal fibres and on the thermal effects of both treated as well as untreated sisal fibre- epoxy resin composites is also looked into.

## **2. EXPERIMENTAL DETAILS**

### **2.1 Materials and Methods**

The substances that are employed in the study comprised: Epolam 2015 Epoxy resin along with Epolam 2014 hardener that have been acquired from AMT composites of South Africa. Sisal fibers at a package of 10 kg are acquired in Mogotio farm, Nakuru County, Republic of Kenya. Sodium Hydroxide was supplied by Minema chemicals (Pty) restricted while 3-glycidyloxypropyltrimethoxy-silane, methanol and hydrochloric acid have been supplied through Sigma Aldrich restricted, all from South Africa.

The sisal fibres have been mercerized by immersion in 5% sodium hydroxide solution for 20 hours. The mercerized fibres are subsequently cleaned using purified water so as to eliminate the sodium hydroxide and then further immersed in 1% acetic acid so as to equalize any balance sodium hydroxide. This were accompanied by immersion in a silane mixture composed of 5% of 3 glycidyloxy-propyl-trimethoxy-silane reduced at a 95% mixture of methanol. The methanols were utilized so as to hydrolyse the silane and ensure it is lively [17]. This therapy has been subsequently succeeded by immersion of these fibres at a 67.5% mixture of hydrochloric acid for 1 hour [29]. The handled fibres are, then, cleaned using de-ionized water and dehumidified in an oven in 45° C over 24 hours.

Sisal fibres for strengthen have been trimmed with a pair of scissors and straightened with a comb to prevent bunching, where could likewise decrease their wetting with resin and so, decrease the effectiveness of strengthening. The fibres have been then evaluated with a technological stability and categorized in various fibre weights for strengthening. The composites have been subsequently forged by utilizing the vacuum infusion technique. The fabricated sisal fibre increased epoxy resin composites were then cured in air for more than 24 hours and subsequently cured in an oven in 80° C for more than 4 hours according to the provider. The strengthening was diverse from 0 to 15 wt%.

### **2.2 Scanning Electron Microscopy (SEM)**

The longitudinal and transverse surface morphologies of the composites at different fibre load fractions for both treated as well as untreated sisal fibre-epoxy resin composites was examined utilizing electron microscopy method.

### **2.3 Thermal analyses**

The following thermal analyses were conducted

#### **2.3.1 Thermo-Gravimetric Analysis (TGA)**

Thermal degradation and stability of the polymer epoxy as well as its composites have been analyzed by Thermo-Gravimetric Analysis (TGA). Thermal gravimetric analyses were carried out to monitor the changes that is taking place in the epoxy resin, treated as well as untreated sisal fibre-epoxy resin composites as a function of exposure to heating. The

thermal stability was measured using a TA instrument; thermal universal analyser, V4.5A. The examinations have been carried out using a nitrogen gas flow at the rate of 100 mL/min from 20°C to 600°C, at a heating run of 10°C per minute.

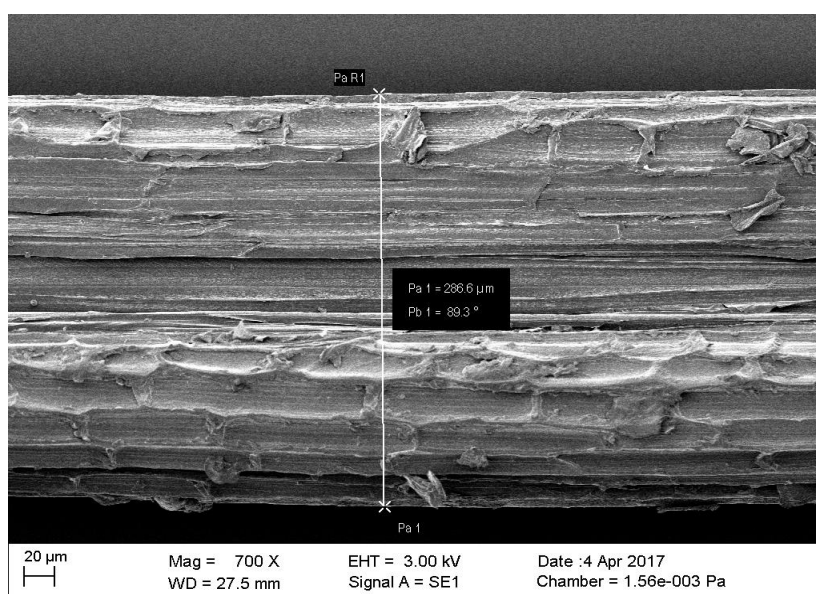
### 2.3.2 Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) quantifications of pure epoxy, treated and untreated sisalfibre– epoxy resin composites samples were performed on a thermal universal analyser V4.5A. The heating rate of 10<sup>0</sup>C/min was utilized beneath dry nitrogen gas in a flow rate of 100 mL/min from 20°C to 600°C.

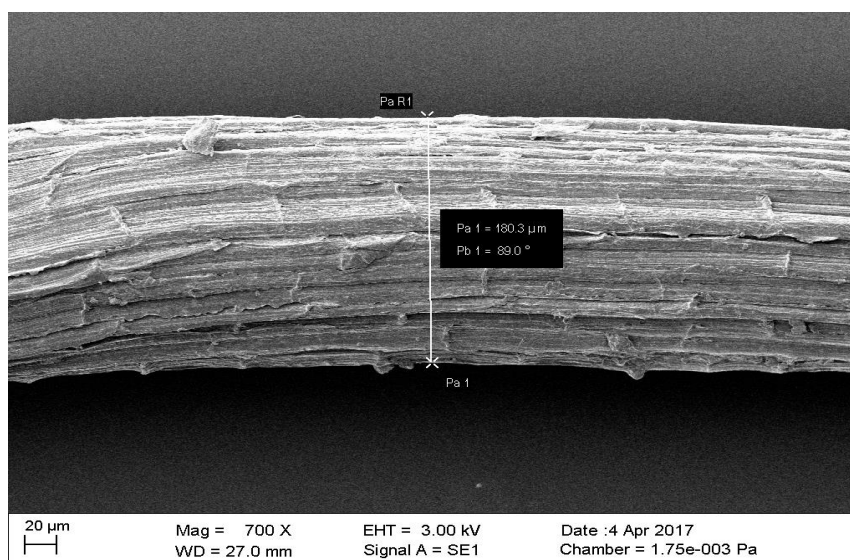
## 3. RESULTS AND DISCUSSIONS

### 3.1 Structure and Morphology

The untreated as well as treated sisal fibres are concerned to scrutinizing electron microscopy along the outcome images such as the ones indicated in figures 1 and 2.



**Figure 1: Untreated sisal Fibre.**



**Figure 2: Treated Sisal Fibres.**

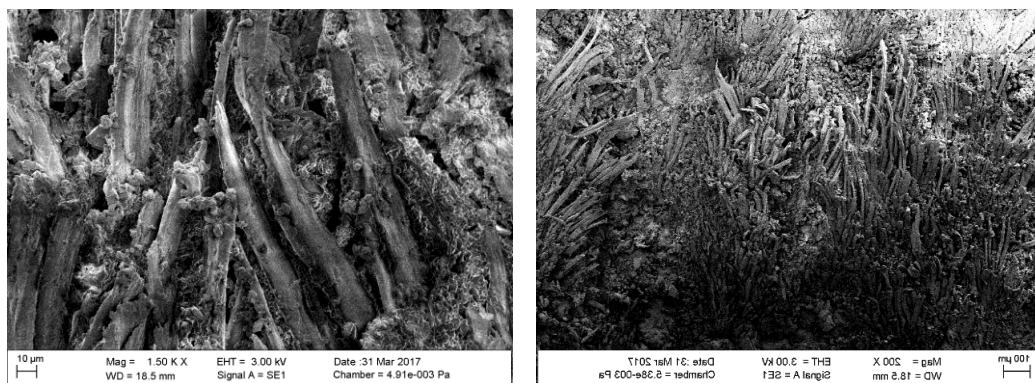


From the images it is obvious that the cross-sectional aspects of the treated sisal fibres were lesser than the untreated sisal fibres. Typically, a decrease in the cross-sectional dimensions of reinforcing fibres leads to higher reinforcing fibre contents and therefore higher mechanical properties were compared to untreated sisal fibres.

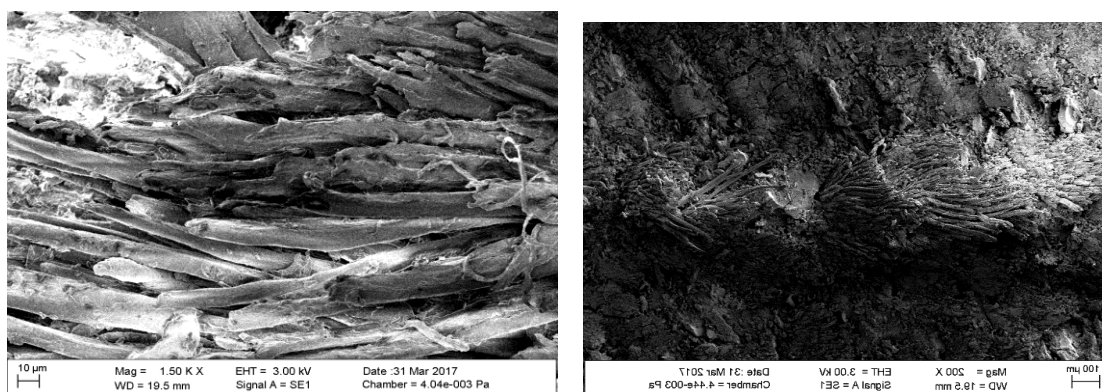
The structure and morphology of both the treated as well as untreated sisal fibre-epoxy resin composites were also determined using Scanning Electron Microscopy (SEM) techniques. The morphology of both the longitudinal and transverse cross sections of the composites at various fibre loadings were investigated using the SEM. Five scans for each fibre loading was taken with representative results and shown in the figures 3 to 9.



**Figure 3: SEM Image of Longitudinal (left) and Transverse (right) cross Section of 5wt% untreated Sisal Fibre-epoxy Resin Composite.**

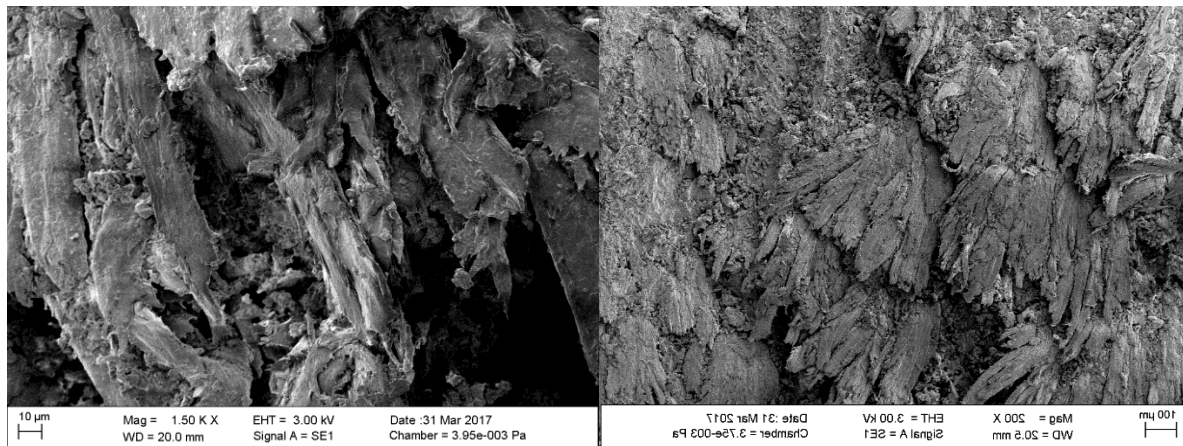


**Figure 4: SEM Image of Longitudinal and Transverse Cross-Section of 10wt% Untreated Sisal Fibre-Epoxy Resin Composite**

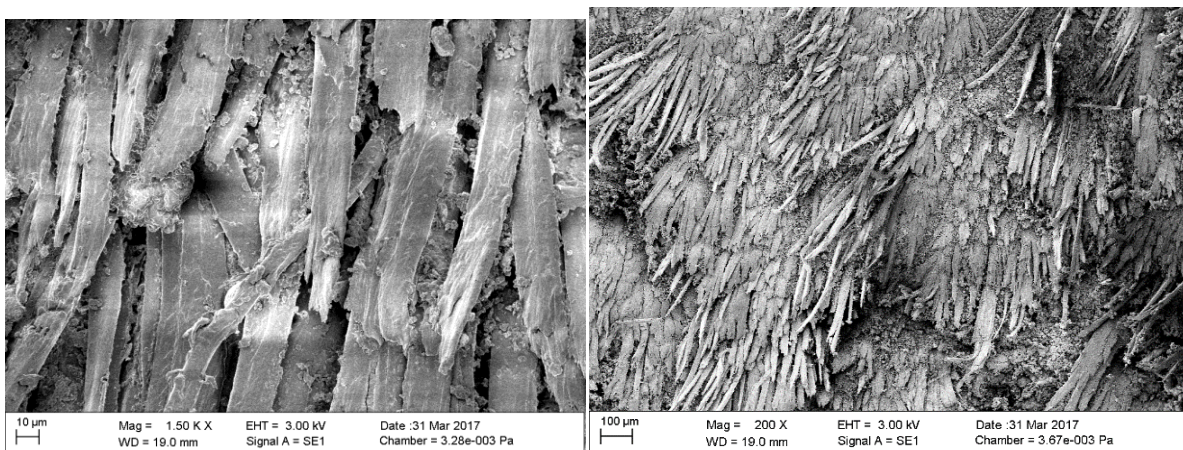


**Figure 5: SEM Image of Longitudinal and Transverse Cross-Section of 15wt% Untreated Sisal Fibre-Epoxy Resin Composites**

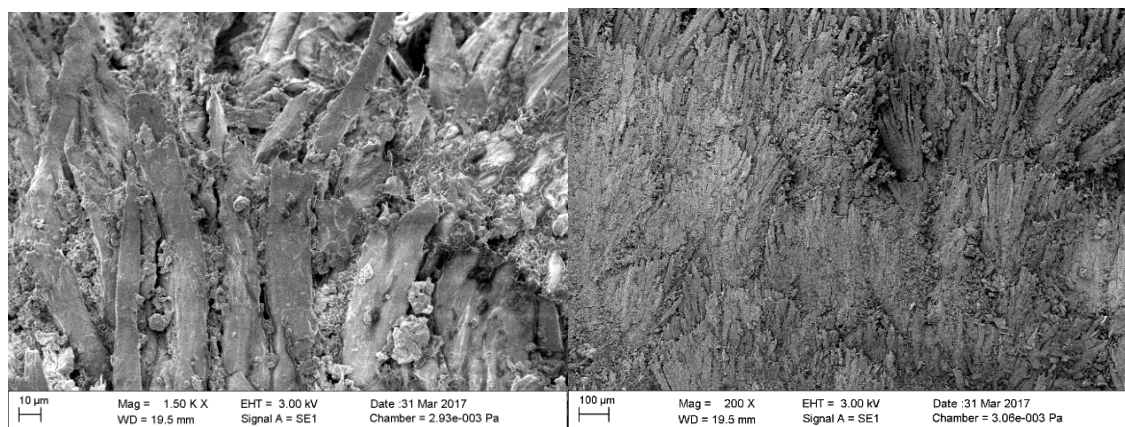




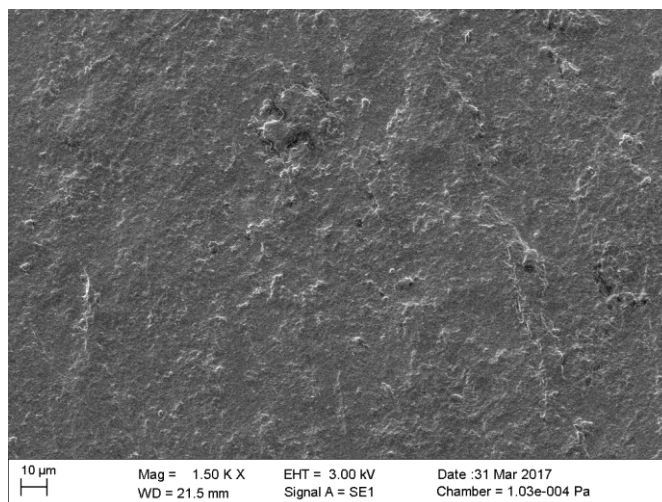
**Figure 6: SEM Image of Longitudinal and Transverse Cross-Section of 5wt% Treated Sisal Fibre-Epoxy Resin composite**



**Figure 7: SEM Image of Longitudinal and Transverse Cross-Section of 10wt% Treated Sisal Fibre-Epoxy Resin composite**



**Figure 8: SEM Image of Longitudinal and Transverse Cross-Section of 15wt% Treated Sisal Fibre-Epoxy Resin Composite.**

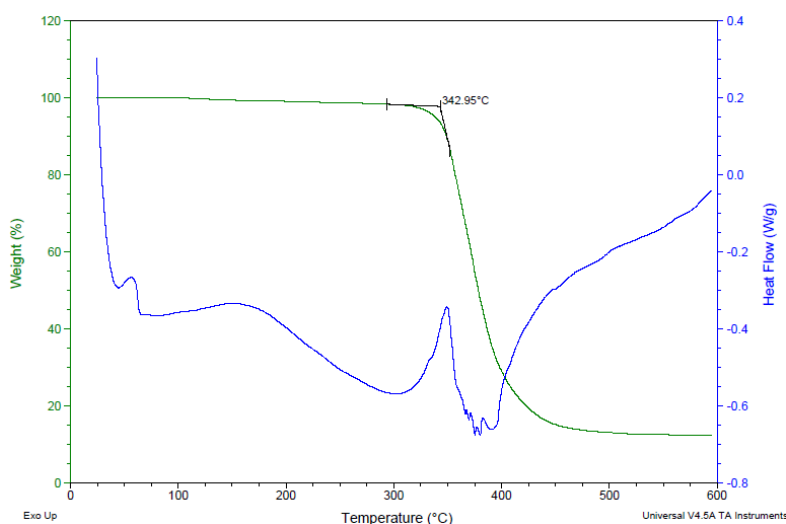


**Figure 9: SEM Image Longitudinal and Transverse Cross-Section of Pure Epoxy.**

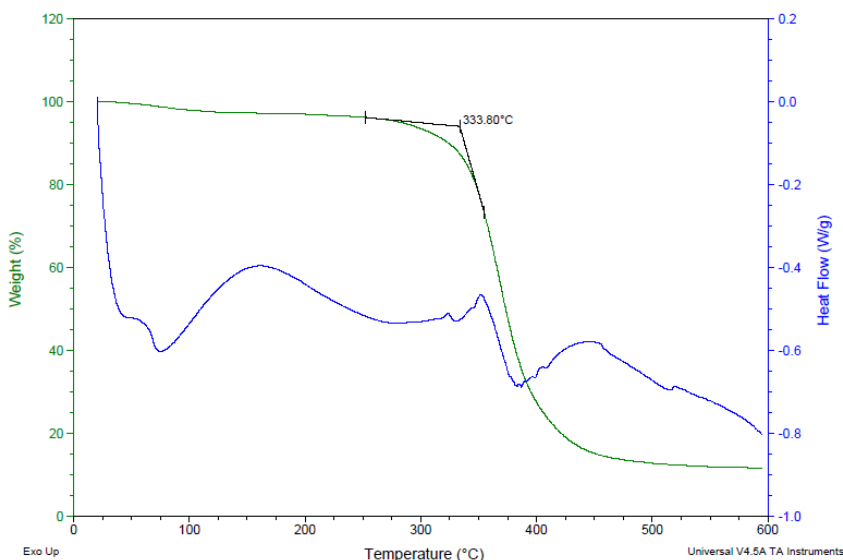
From an examination of the micrographs, it is evident that there is aggregation of the untreated sisal fibres, within the epoxy resin as opposed to the case for treated sisal fibre at a fibre weight fraction of 10% and the converse at fibre weight fractions of 5% and 15%. Further, from an investigation of the fibre-matrix interface, it is evident that there is an increase in bonding area in the treated sisal fibre-epoxy resin composites as compared to the case for the untreated sisal fibre-epoxy resin composites. This difference is considered to be the solution for treatment of the fibre surface with alkali, silane and acid hydrolysis which is known to reduce their hydrophilicity resulting into improved adhesion connecting them and the hydrophobic matrices. Fibre treatment causes the silanols in the silane to be grafted onto the hydroxyl group of the fibres resulting in reduced moisture uptake (17). This allows for better adhesion between the treated fibres along with the hydrophobic matrix in contrast to the case of the untreated fibres and matrix (8).

### 3.2 Thermal Analyses

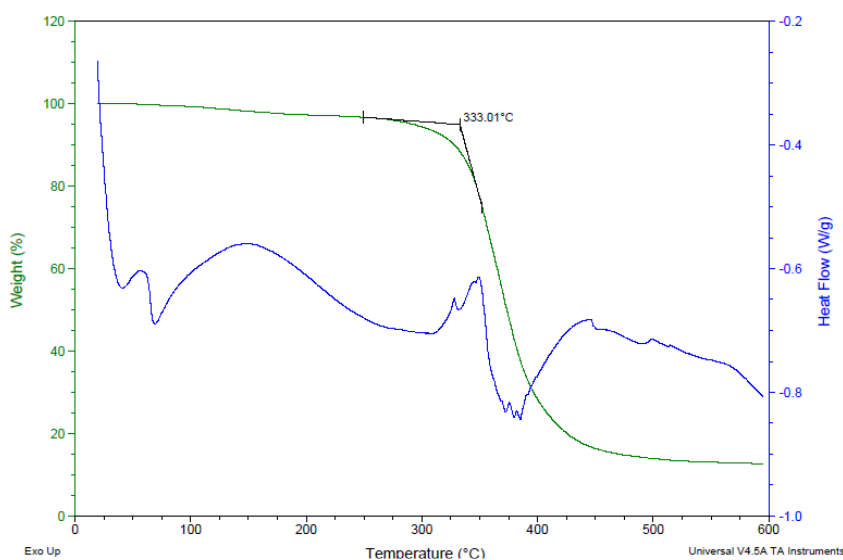
Both Thermal Gravimetric Analyses (TGA) and the Dynamic Scanning Calorimetry (DSC) for clean epoxy resin, treated as well as untreated sisal fibre-epoxy resin composites were investigated. The curves for thermal gravimetric analyses and the curves for dynamic scanning calorimetry for each of the samples tested were plotted on the same graph.



**Figure 10: Pure Epoxy Resin.**



**Figure 11: Untreated Sisal Fibre-epoxy Resin Composite.**



**Figure 12: Treated Sisal Fibre-Epoxy Resin Composites.**

### Key

- Thermal gravimetric analyses curve
- Dynamic scanning calorimetry curve

The curves in Figures 10, 11 and 12 exhibit three sections: initial flat sections, then sections with constant slopes and finally flat sections. In the initial flat sections, there was a weight loss for the epoxy resin, untreated sisal fibre-epoxy resin composite and the treated sisal fibre-epoxy resin composite samples of the order 5%, 10% and 10%, respectively, as seen in the three respective figures. In the sections with constant slopes, there was a weight loss of 80%, 75% and 75%, respectively, for epoxy resin, untreated sisal fibre-epoxy resin composites and the treated sisal fibre-epoxy resin composites, as seen in the three respective figures. In the final flat sections, all the mass degraded and all that was left



behind was a black carbonaceous residue known as char. The initial weight loss of all samples began at temperatures of between 25°C and 30°C and the behaviour of degradation was observed up to 600°C. The initial degradation observed was because of the evaporation of moisture from the samples. All the samples of the pure epoxy resin, untreated sisal fibre-epoxy resin composites and the treated sisal fibre-epoxy resin composites moved to full degradation at temperatures of between 400°C and 475°C. The complete degradation in the fibre composites occurred when all the cellulose fully degraded. Full degradation in the three figures is evident by the sudden levelling of the graphs.

In the Dynamic Scanning Calorimetry (DSC) plots, all exhibited a glass transition temperature of between 50°C and 75°C. Further, all the samples exhibited a melting temperature of between 350°C and 400°C. The first peaks of the DSC curve of epoxy resin, untreated sisal fibre-epoxy resin composite and treated sisal fibre-epoxy resin composites in Figures 10, 11 and 12 correspond to a mass losses of 55%, 60% and 65%, respectively. This is as a result of the loss of moisture. The remaining material in the epoxy resin, untreated sisal fibre-epoxy resin composites and treated sisal fibre-epoxy resin composites is seen in the curves to have remained thermally stable up to temperatures of 325°C, 350°C and 350°C, for epoxy resin, untreated sisal fibre-epoxy resin composites and treated sisal fibre-epoxy resin composites, respectively. Thereafter the rates of pyrolysis reduced gradually until they reached minimum values at 400°C, 380°C and 390°C, for epoxy resin, untreated sisal fibre –epoxy resin composites and treated sisal fibre-epoxy resin composites, respectively. From 400°C to 600°C, pyrolysis continued progressively at very low rates. The first peaks in the DSC plots are due to pyrolysis of hemicelluloses, while the second peaks in the DSC plots are attributed to the pyrolysis of cellulose and lignin (6). The general behavior of the samples exposed to Dynamic Scanning Calorimetry can be explained by a decrease in the cohesive energy in the samples due to the rupture of the hydrogen bonds as a result of heating.

#### 4. CONCLUSIONS

- There is a reduction in the size of the lumen and cell wall after the fibre treatment.
- There is a reduction in the dimensions of the fibres after treatment.
- There is evidence of increase in surface roughness of the reinforcing fibres.
- There is evidence of differences in the TGA and DSC curves as a result of fibre treatment.

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## AUTHORS PROFILE



**Wilson Webo**, is a researcher and a Lecturer of Mechanical Engineering with specialization in Composite Materials. He has written 6 conference papers and journal articles. He is currently a doctoral candidate in Mechanical Engineering Department at the Vaal University of Technology, South Africa, having successfully completed his Masters Degree in Mechanical Engineering from the same institution. His undergraduate degree was in Mechanical and Production Engineering from Moi University, Kenya. He is also a registered engineer with the Engineers Board of Kenya.



**Professor LM Masu**, holds BSc (Hons) Mech.Eng, MSc. Mech.Eng., PhD. (Leeds), PGDip. B Admin (UDW) and MBA (UFS). He is registered as Pr.Eng (ECSA), FSAIMEchE, R Eng (K) and MIEK. Prof Masu has published 51 journal and 33 conferences articles, co-authored 1 book and 1 book chapter. He has a total of 38 years of academic experience, of which 27 years has been on academic managerial levels. In addition to this wealthy experience, Prof Masu has 4 years hands on industrial experience.



**Maina Maringa**, is a Professor of Mechanical Engineering of the Technical University of Kenya (TUK) with specialisation in the fields of composite materials and stress analysis. He has read and written 70 conference papers and journal articles, three university level books and 1 tertiary level book. Maina has over the years served as an editor in Chief and referee for 17 international refereed journals and numerous conferences. He has over the last 29 years taught in 5 universities and one tertiary level institution in 4 countries worldwide. Maina joined the Central University of Technology (CUT), Free State, South Africa in the year 2016 where he has and is engaged in research and supervision of postgraduate students on additive manufacturing of metals, polymers and composites at the Centre for Rapid Prototyping and Manufacturing (CRPM).